

**PARTICLE SIZE DISTRIBUTION DATA FOR
LIBBY AMPHIBOLE STRUCTURES OBSERVED IN AIR
AT THE LIBBY ASBESTOS SUPERFUND SITE**

July 14, 2010

**Prepared by:
U.S. Environmental Protection Agency
Region 8
Denver, CO**



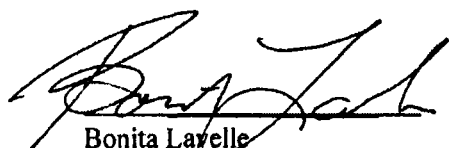
With Technical Assistance from:

**SRC, Inc.
Denver, CO**



APPROVAL PAGE

This report, *Particle Size Distribution Data for Libby Amphibole Structures Observed in Air at the Libby Asbestos Superfund Site*, is approved for distribution.


Bonita Lavelle
U.S. EPA, Region 8

7/19/10
Date

**PARTICLE SIZE DISTRIBUTION DATA FOR
LIBBY AMPHIBOLE STRUCTURES OBSERVED IN AIR
AT THE LIBBY ASBESTOS SUPERFUND SITE**

1.0 INTRODUCTION

Libby is a community in northwestern Montana that is located near a large open-pit vermiculite mine. Vermiculite from this mine contains varying levels of a form of asbestos referred to as Libby Amphibole (LA). In 1999, EPA Region 8 initiated environmental investigations in the town of Libby and in February, 2002, EPA listed the Libby Asbestos Site (the Site) on the National Priorities List. The Site includes the former vermiculite mine and residential homes, commercial businesses, schools and parks that may have become contaminated with asbestos fibers as a result of vermiculite mining and processing conducted in and around Libby as well as other areas in the vicinity that may have been impacted by mining-related releases of asbestos. Historic mining, milling, and processing operations at the Site, as well as bulk transfer of mining-related materials, tailings, and waste to locations throughout Libby Valley, are known to have resulted in releases of vermiculite and LA to the environment.

As part of the response actions taken pursuant to the Comprehensive Environmental Response, Compensation and Liability Act, EPA has performed a number of investigations to characterize the nature and extent of LA contamination of air, soil, dust and other media in and around the community of Libby. Because available information suggests that the toxicity of asbestos is at least partially influenced by the size of the inhaled asbestos particles, these investigations have included the measurement of the dimensions (length and width) of LA particles observed in samples collected from the Libby site.

The purpose of this report is to summarize size distribution data for LA particles that have been observed in air samples collected at the site, and to utilize these data to make comparisons between various subsets of the data to determine if any important differences in particles size distributions can be recognized.

2.0 METHODS

2.1 Data Overview

EPA has been collecting samples of air since 2001 at the Libby site. Table 1 provides an overview of the sampling programs that have generated these data. The raw data for the air samples included in this assessment are provided in Appendix A.

Most of the samples that have been collected have been analyzed for asbestos by transmission electron microscopy (TEM) using either ISO 10312 (ISO 1995) or AHERA (AHERA 1986)

counting rules, as modified by site-specific modifications as described in modifications forms LB-000016 and LB-000031 (provided in Appendix B). In all cases, the data that are recorded during the analysis of a sample include the length, width and aspect ratio (length/width) of all particles that meet the counting rules specified for the analysis.

2.2 Data Presentation

One convenient method for comparing the size distributions of two different sets of LA particles is through a graph that plots the cumulative distribution function (CDF) for each particle set. This graphical format shows the fraction of all particles that have a dimension less than some specified value. This format is used in this document to present the distributions of length, width and aspect ratio.

There are a number of statistical tests that can be used to compare two distributions in order to support a statistical statement about whether the distributions are “same” or “different”. Such comparisons are complicated by the fact that the distributions may be similar over some intervals and dissimilar over other intervals. However, at present, data are not sufficient to know which parts of the distribution are most important from a toxicological perspective. Therefore, this document relies upon simple visual inspection to assess the degree of difference between various regions of differing distributions.

3.0 RESULTS

3.1 Data Validation

The Libby2 database and Libby OU3 database have a number of built-in quality control checks to identify unexpected or unallowable data values during upload into the database. Any issues identified by these automatic upload checks were resolved by consultation with the analytical laboratory before entry of the data into the database. After entry of the data into the database, several additional data verification steps were taken to ensure the data were recorded and entered correctly. A total of 29,504 LA structures are included in Table 1. Of these structures, 25% have undergone data validation in accord with standard site-wide operating procedures (USEPA 2008b) to ensure that data for length, width, particle type, and mineral class are correct. Of the structures that have undergone validation, only 39 of 7,464 (0.5%) structures had errors in length, width, or mineral class. These errors were corrected and the database updated as appropriate.

3.2 Consolidated Data Set

Originally, most samples of air at Libby were analyzed using a counting rule based on a fiber aspect ratio of 5:1. More recently, most air samples are counted using an aspect ratio rule of 3:1. Because this rule has varied over time, Libby-specific laboratory modifications LB-000016 and

LB-000031 (see Attachment B) were created to document the historic modifications and instructions that laboratories have followed throughout the Libby program.

Figure 3-1 presents the particle size distributions for 29,504 LA particles observed to date¹ in air samples collected at the Libby Asbestos Superfund site that have an aspect ratio of 5:1 or more, along with the distributions for 11,451 particles that were counted using an aspect ratio rule of 3:1. As seen, the distributions are very similar. This is because the number LA particles that have an aspect ratio > 3:1 and < 5:1 is a relatively small fraction of the total (7%).

For simplicity, all remaining analyses focus on the set of particles with an aspect ratio of 5:1 or more.

3.3 Frequency of Complex Structures

Asbestos particles occur not only as fibers but also in more complex structures including bundles, clusters, and matrix complexes. The frequency of these structure types in air samples from Libby are summarized below:

Type ²	Number	Frequency
Fiber	23,933	81%
Bundle	2,366	8%
Matrix	3,150	11%
Cluster	54	0.2%
Total	29,504	100%

As shown, most (81%) of the enumerated structures are fibers, with less than 20 % complex structures.

3.4 Comparisons of Stratified Data Sets

The data sets shown in Figure 3-1 are based on air samples that were collected at a number of different locations around the site, and which were analyzed by several different methods. In order to investigate whether there are any important differences in size distributions between operable units, sampling locations (indoor, outdoor), activity (e.g., active or passive), and /or analytical method, the consolidated data set was partitioned into a number of subsets, as follows:

¹ Based on a query of the Libby2 database on 12/08/09 and the Libby OU3 database on 2/9/10.

² In some cases, the structure type assignment provided by the laboratory was not a valid choice according to the recording rules for the specified analysis method. Table A-1 in Appendix A presents the types of invalid structure types and the structure class assumption that was made in order to include the structure in this report.

Figure	Comparison
3-2	LA particles observed in air stratified by structure type
3-3	LA particles observed in air stratified by Operable Unit
3-4	LA particles observed in air stratified by sample type (ambient, indoor, outdoor ABS)
3-5	LA particles observed in air stratified by preparation method (direct vs indirect)
3-6	LA particles observed in air stratified by analysis method (ISO vs AHERA)

Figure 3-2 is a comparison of different structure types (fiber, bundles, and matrices). Clusters were not included because there were too few for a distribution to be meaningful. As seen, the length distribution for matrix particles is somewhat left-shifted compared to fibers. This is perhaps expected because some portion of the fiber length in matrix fibers is obscured by the matrix particle. In contrast, the length and thickness distributions for bundles are right-shifted compared to fibers. This is expected because a bundle is several fibers lying in parallel.

Figure 3-3 compares the size distributions of LA at different operable units (OUs) at the site. As seen, there appears to be little difference in structures from the different OUs.

Figure 3-4 shows the distribution of structure sizes for different types of air samples. Samples have been placed into three groups: ambient air, indoor ABS, and outdoor ABS. As shown, the length and width distributions for indoor and outdoor ABS samples are relatively similar, while the length and width distribution for ambient air samples appear to be right shifted. However, this observation should be considered to be relatively uncertain because of the small number (136) of particles that constitute the ambient air data set.

Figure 3-5 compares the size distributions for samples using direct and indirect preparation methods. As shown, there is little difference in the distributions or either length or width, suggesting that preparation method does not have a significant impact on particle size.

Figure 3-6 compares the particle size distributions as a function of analytical counting rules. As shown, the length and width distributions for particles analyzed using AHERA rules tend to be somewhat right-shifted relative to the distributions for particles analyzed using ISO 10312 rules. This apparent difference might be related either to differences in counting rules between methods, or possibly to differences in the nature of samples analyzed by each method. In either event, the difference between methods appears to be relatively small.

4.0 SUMMARY

Particle size data are available for nearly 30,000 LA structures that have been observed in air samples collected at the Libby Asbestos Superfund site. Most (about 80%) LA particles are fibers, with less than 20% complex structures (bundles, clusters, or matrices). LA particle

lengths typically range from a little less than 1 μm up to 20-30 μm , and occasionally higher. The average length is about 7 μm . Thicknesses typically range from about 0.1 μm up to about 2 μm , with an average of about 0.5 μm . Although some variations occur, particle size distributions are generally similar between different locations and between different types of samples.

5.0 REFERENCES

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USEPA. 2007c. Sampling and Analysis Plan for Activity-Based Indoor Air Exposures, Operable Unit 4, Libby, Montana, Superfund Site. Final. U.S. Environmental Protection Agency, Region 8. July 6, 2007.

USEPA 2008a. Request for Modification to Laboratory Activities LB-000031A. Requested by Lynn Woodbury of Syracuse Research Corporation. January 18, 2008.

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Table 1. Air Sample Collection Programs

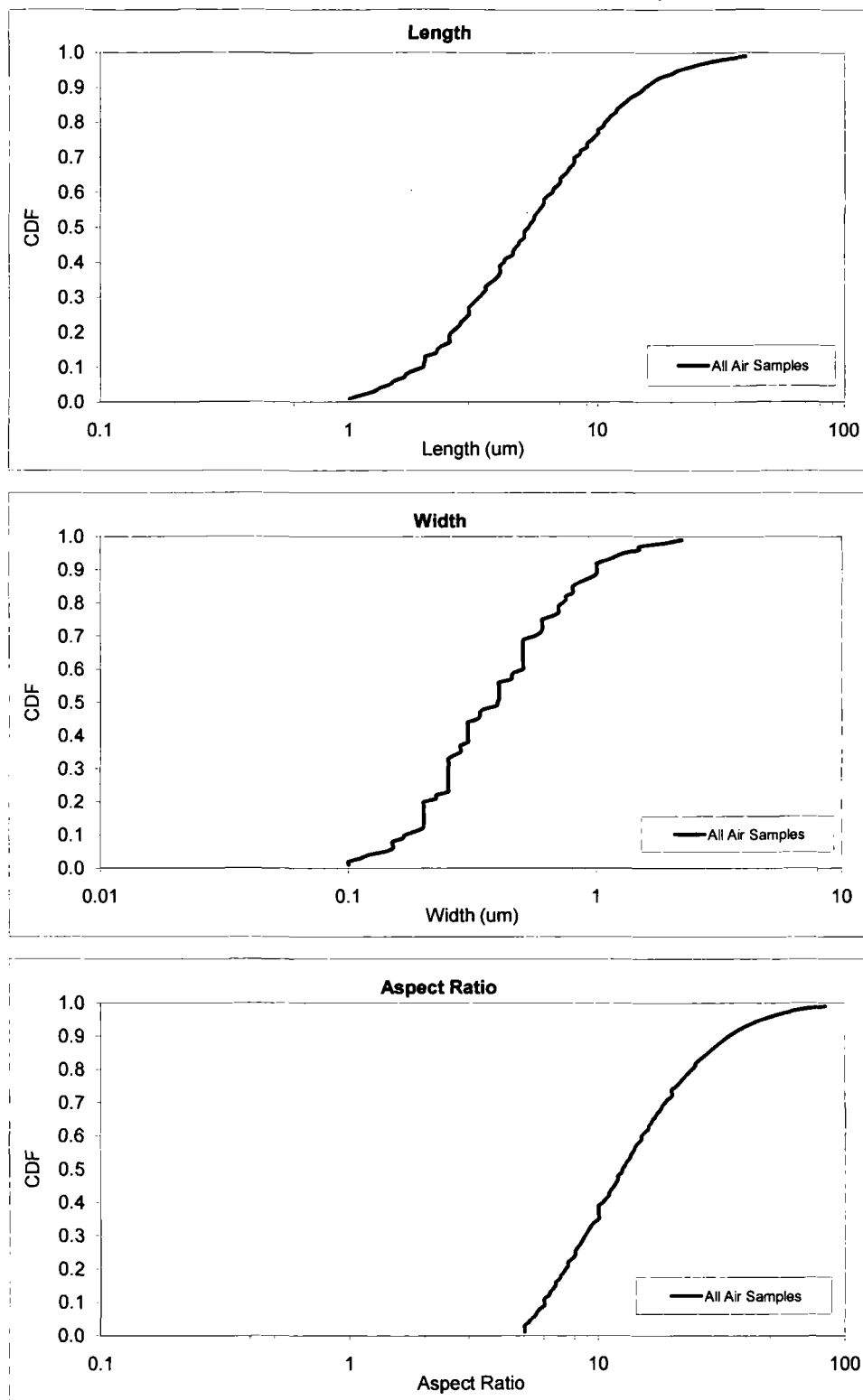
Program	Program Description	Program Date Range	Sampling and Analysis Plan (s)	Number of LA Structures ^(a)
Phase 1	Initial investigation sampling to assess nature and extent of potential contamination. Includes source areas (e.g., screening plant, export plant), commercial buildings, and residential properties.	Dec 1999 - present	USEPA 2000	328
Phase 1R	Monitoring and confirmation sampling as part of clean-up activities.	Jun 2000 - present	USEPA 2000	18,525
Phase 2	Activity-based sampling (ABS) included four scenarios: 1) routine indoor activities, 2) active cleaning, 3) simulated remodeling disturbances, 4) garden rototilling.	Mar - Nov 2001	USEPA 2001	867
Phase 2R	Monitoring and confirmation sampling as part of Phase 2	Apr 2008 - Nov 2009		1,717
CSS	Contaminant Screening Study of Libby properties to determine need for remediation.	Apr 2003 - Oct 2006	USEPA 2002	3
SQAPP	Sampling to address risk assessment data gaps. Included indoor ABS (routine activities) and outdoor ABS (raking, mowing, playing), as well as clean-up evaluation samples.	Jun 2005 - Oct 2006	USEPA 2005	1,456
Ambient Air (AA)	Ambient air monitoring program for 14 stations in OU4, 2 stations in OU2, 2 stations in OU6. Samples represent long-term (continuous 5-day) collection periods.	Oct 2006 - Jun 2008	USEPA 2006, USEPA 2007a	136
OU4 Indoor/Outdoor ABS	Sampling to assess exposures during indoor ABS (passive & active activities) and outdoor ABS (raking, mowing, playing) in OU4.	Jul 2007- Jun 2008	USEPA 2007b, USEPA 2007c	5,603
Indoor Schools	Stationary air sample collection from within Libby public schools	Dec 2008	USEPA 2008d	2
Outdoor Schools	Outdoor ABS sampling from Libby public schools simulating exposures to students and maintenance staff.	Jul - Sept 2009	USEPA 2009b	5
Phase 2 (OU3)	Ambient air sampling. Samples represent long-term (continuous 5-day) collection periods.	July - Oct 2008	USEPA 2008c	67
Phase 3 (OU3)	ABS air sampling of ATV riding, hiking, camp fire construction	Aug - Nov 2009	USEPA 2009a	59
Clean-up Evaluation	Sampling to monitor air and dust levels after completion of clean-up activities at 31 properties.	Nov 2003 - Feb 2004	USEPA 2003	5
Other	Includes various site-specific sampling investigations (e.g., Stimson Lumber, Flyway, BNSF) and smaller-scale sampling programs.	Aug 2001 - present	various	731

(a) Restricted to LA structures recorded in accordance with a 5:1 aspect ratio rule.

LA structure counts are based on a download of Libby 2DB performed on 12-8-09 and the Libby OU3 DB on 2-9-10.

Other		
Program	LA Structures	Description
1A	9	AIRS Site (418 Mineral Ave)
BN	17	BNSF
CR	3	Cumulative Risk Study
DM	1	Demolition Sampling from 2006 only
E1	1	BNSF Rail Yard Exclusion Zones
EP	104	Export Plant
FC	184	Flower Creek
FL	146	WR Grace (Flyway site)
SL	266	Stimson Lumber

Figure 3-1. Particle Size Distributions of LA Particles in Libby Air Samples

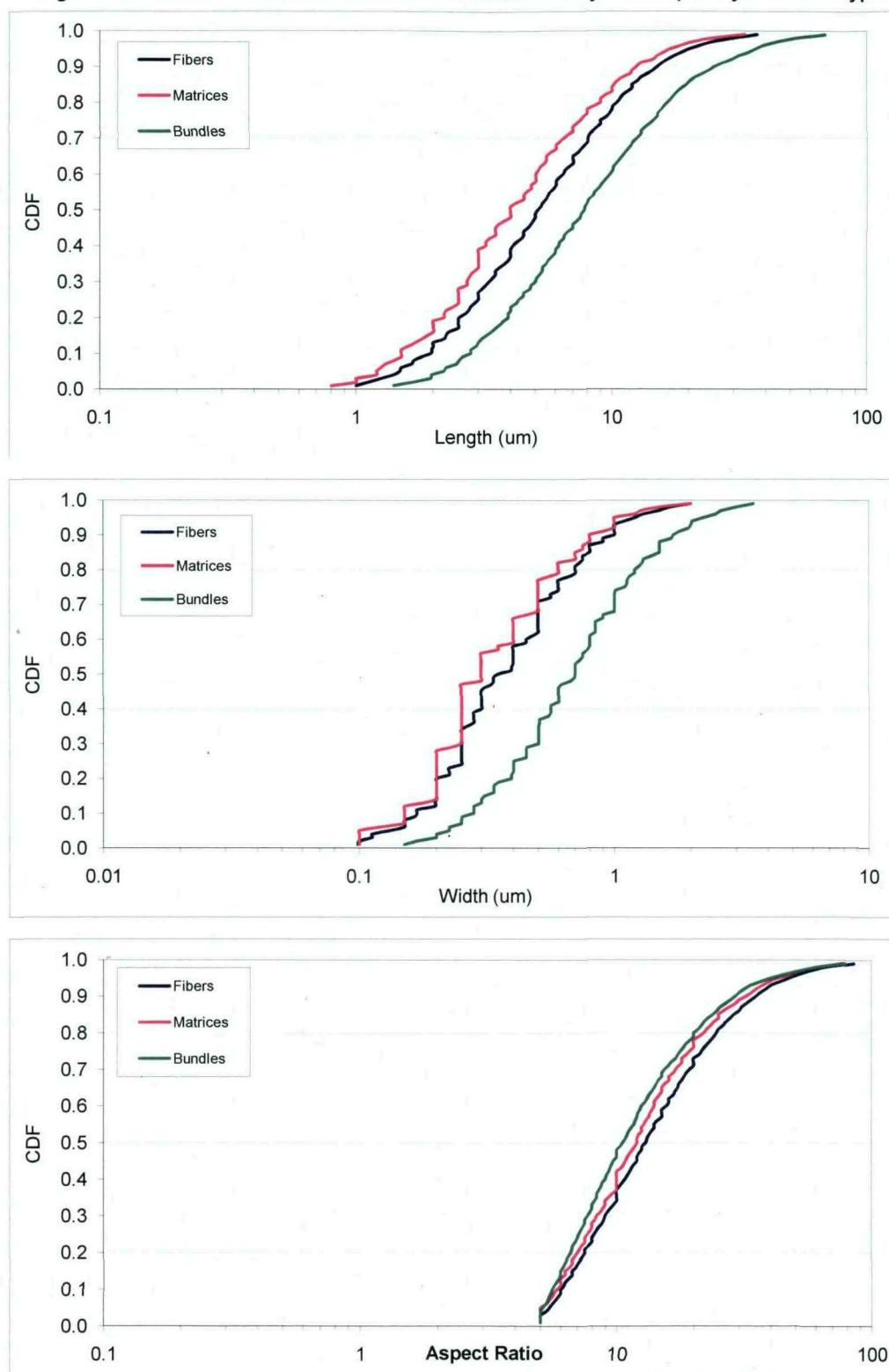


Data are based on a download of Libby 2DB performed on 12-8-09 and the Libby OU3 DB on 2-9-10.

All Air Samples

Number of Structures (29,504)		
Type	Number	Frequency
F	23,933	81%
B	2,366	8%
M	3,150	11%
C	54	0.2%

Figure 3-2. Particle Size Distributions of LA Particles in Libby Air Samples by Structure Type

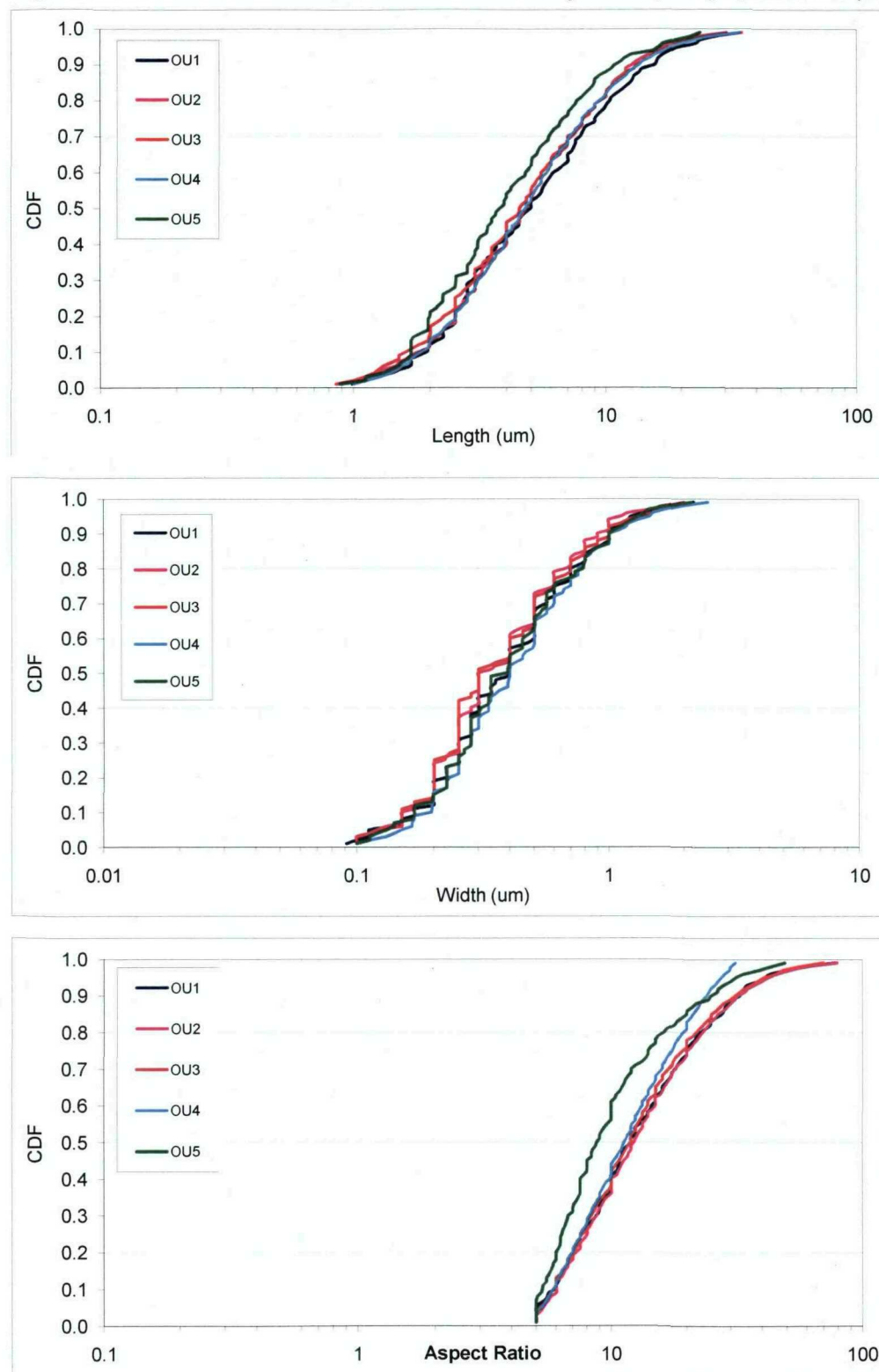


Data are based on a download of Libby 2DB performed on 12-8-09 and the Libby OU3 DB on 2-9-10.

Structure Type	N Structures
F	23,933
B	2,366
M	3,150

Clusters have not been included in this figure because N = 54 and this is not believed to be a sufficient number of structures.

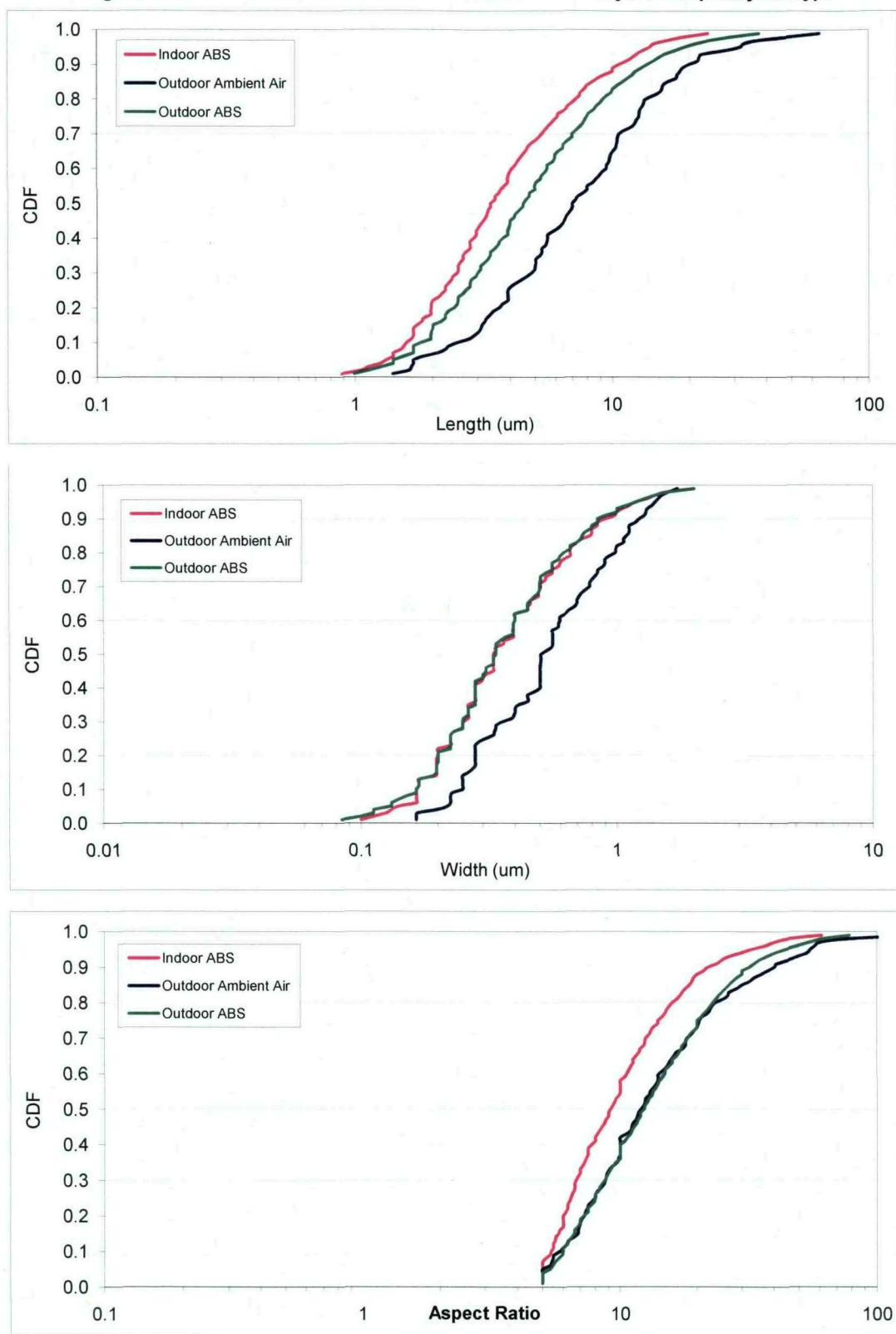
Figure 3-3. Particle Size Distributions of LA Particles in Libby Air Samples by Operable Unit (OU)



Data are based on a download of Libby 2DB performed on 12-8-09 and the Libby OU3 DB on 2-9-10.

OU	N Structures
1	447
2	7,421
3	4,382
4	13,005
5	335

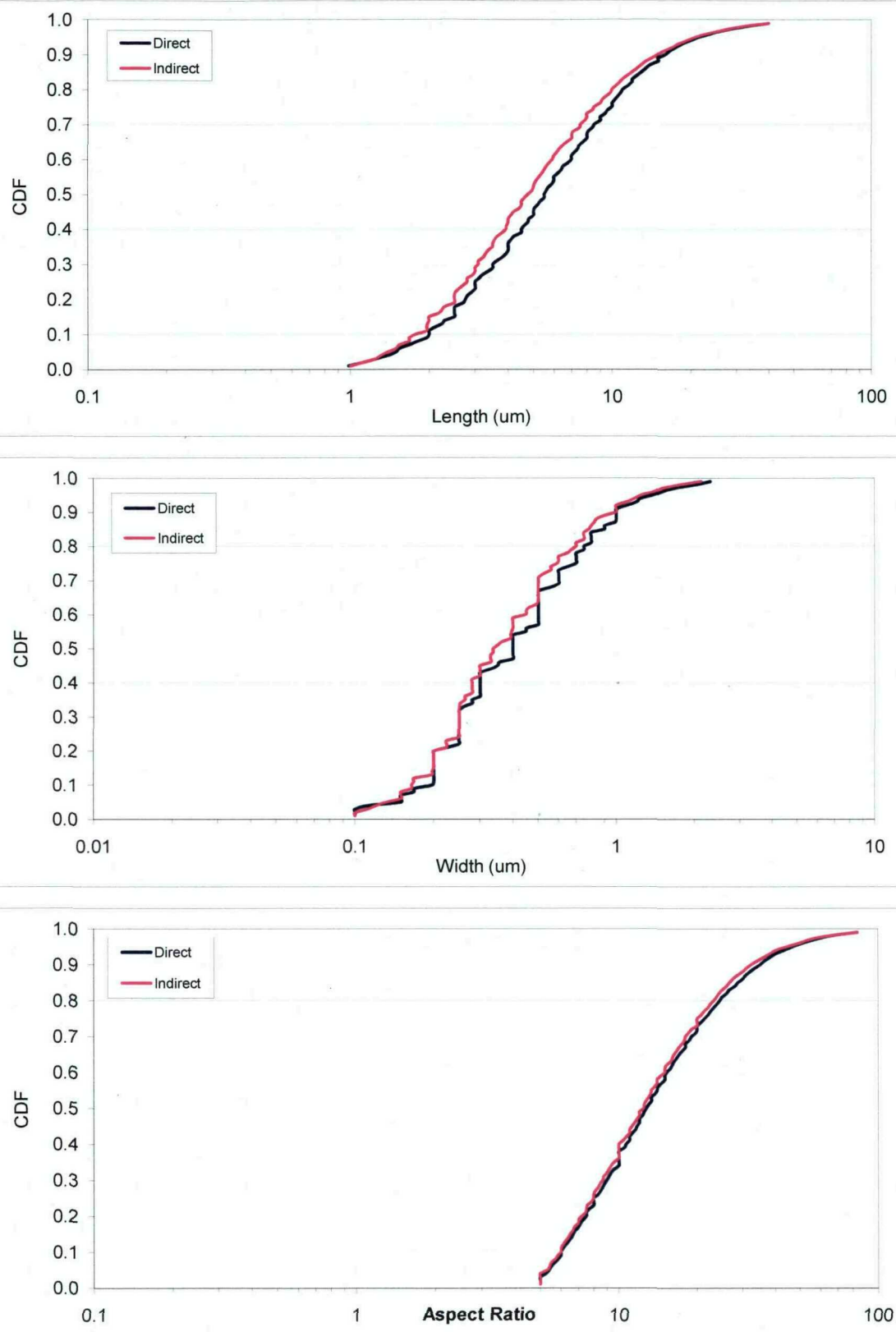
Figure 3-4. Particle Size Distributions of LA Particles in Libby Air Samples by Air Type



Data are based on a download of Libby 2DB performed on 12-8-09 and the Libby OU3 DB on 2-9-10.

Samples Source	N Structures
Ambient Air	136
Indoor ABS	891
Outdoor ABS	5,953

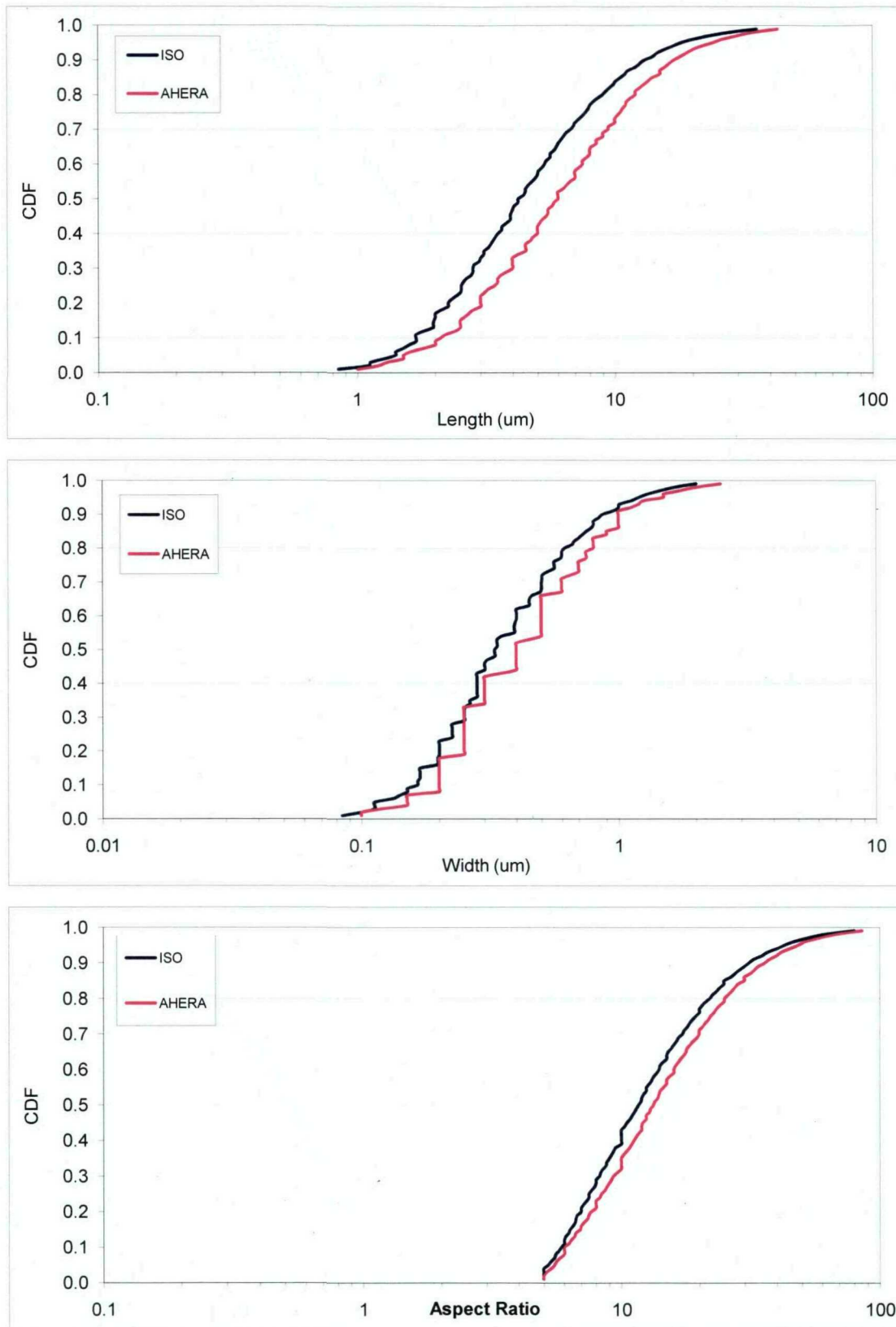
Figure 3-5. Particle Size Distributions of LA Particles in Libby Air Samples by Preparation Method



Data are based on a download of Libby 2DB performed on 12-8-09 and the Libby OU3 DB on 2-9-10.

Preparation	N Structures
Direct	17,578
Indirect	11,926

Figure 3-6. Particle Size Distributions of LA Particles in Libby Air Samples by Analysis Method



Data are based on a download of Libby 2DB performed on 12-8-09 and the Libby OU3 DB on 2-9-10.

Analysis Method	N Structures
ISO	12,657
AHERA	16,847

APPENDIX A

RAW DATA: LA STRUCTURE DATA FROM THE LIBBY 2 DATABASE AND THE LIBBY OU3 DATABASE

Libby2DB based on a download date of 12/8/09
Libby OU3 DB based on a download date of 2/9/10

See attached file

Table A-1. Structure Type Assignments

Counting Rule	Structure Type	Structure Class (a)	Fiber?	Notes
AHERA/ASTM	B	B		
	C	C		
	F	F	Y	
	M	(F)	Y	
	MD	(F)	Y	**recorded using ISO structure type, assumed to be disperse matrix
	MD11	(F)	Y	**recorded using ISO structure type, assumed to be disperse matrix
	MF	(F)	Y	**recorded using ISO structure type, assumed to be matrix w/fiber protrusion
ISO 10312	B	B		
	C	C		**recorded using AHERA structure type, assumed to be compact cluster
	CB	B		
	CF	F	Y	
	DM10	F	Y	**assumed to be fiber within a matrix (MF)
	F	F	Y	
	FM	F	Y	**assumed to be fiber within a matrix (MF)
	M	M		**recorded using AHERA structure type, assumed to be compact matrix
	MB	B		
	MB10	B		**assumed to be bundle within a matrix (MB)
	MC	M		**assumed to be a compact matrix
	MC+0	M		
	MC0	M		**assumed to be a compact matrix
	MC10	M		**assumed to be a compact matrix
	MD	F	Y	**assumed to be fiber within a matrix (MF)
	MD10	F	Y	**assumed to be fiber within a matrix (MF)
	MD11	F	Y	**assumed to be fiber within a matrix (MF)
	MF	F	Y	
	MF1	F	Y	**assumed to be fiber within a matrix (MF)
	MF10	F	Y	**assumed to be fiber within a matrix (MF)
	MF2	F	Y	**assumed to be fiber within a matrix (MF)
	ND10	F	Y	**typo MD10; assumed to be fiber within a matrix (MF)

** Structure Type is not valid

(a) For matrices recorded by AHERA counting rules, assumed that all matrices had fiber protrusions.

APPENDIX B

LIBBY-SPECIFIC LABORATORY MODIFICATION FORMS

LB-000016

LB-000031



Request for Modification
to
Laboratory Activities
LB-000016A

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Lynn Woodbury Title: Technical Consultant
Company: Syracuse Research Corporation Date: April 10, 2008

Description of Modification:

Permanent modifications and clarifications to the Transmission Electron Microscopy analysis of air and dust samples using ISO 10312. The purpose of the attached is to document historic modifications & clarifications, and provide additional permanent clarifications.

Reason for Modification:

To optimize the efficiency of air and dust sample analysis and to provide consistency in analytical procedures and data recording in the project laboratories.

Potential Implications of this Modification:

Modifications reflect changes necessary to clarify ISO requirements in relation to project-specific issues. Negative implications - comparisons of the Total # of LA structures between historical results and current results may be biased (high or low) due to differences in recording rules with regard to aspect ratio criteria. Positive implications - consistency in procedures between and within project laboratories and documentation of those procedures.

Laboratory Applicability (circle one): All Individual(s) _____

This laboratory modification is (circle one): **NEW** APPENDS to _____ SUPERCEDES LB-000016

Duration of Modification (circle one):

Temporary Date(s): _____

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable Reject Low Bias Estimate High Bias No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

See attached sheets for the description of the TEM-ISO clarifications/modifications.

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

ISO 10312 MODIFICATIONS AND CLARIFICATIONS

1. Overloading Modification:

The ISO method requirement states that if the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. A rejection criteria of >25% obscuration and <50% intact grid openings will be used for this project. The 25% overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, Camp Dresser McKee, Volpe Center, and Reservoirs.

2. Indirect Preparation of Air Samples Modification:

ISO 10312 is a direct preparation method. If the sample is visibly overloaded or contains loose debris, it will be prepared indirectly according to procedures provided in SOP EPA-Libby-08. Secondary filters will be analyzed according to the ISO counting rules for this project. Calculations will be adjusted to contain a dilution factor. This indirect preparation procedure will enable the capture of data from samples that otherwise would be rejected.

3. Stopping Rule Clarification:

Stopping rules for ISO analyses are completion of the grid opening on which the 100th asbestos structure has been recorded, or a minimum of four grid openings. For this project, a maximum of ten grid openings will be read unless specifically instructed otherwise.

4. Abundant Chrysotile Modification:

If abundant chrysotile is present, the chrysotile count may be terminated in accordance with the counting rules specified in LB-000039.

5. Structure Counting and Recording Modifications and Clarifications:

- a. Non-asbestos material (NAM) structures are not being recorded, unless identified as a "close call" (see LB-000066 for details). This project-specific modification stems from the need only to quantify levels of contaminants of concern (i.e., asbestos) at a given sample location.
- b. Recording rules will be as described in the ISO method except that the aspect ratio requirement will depend upon the classification of the sample as "investigative" or "non-investigative", as specified in LB-000053. If samples are classified as investigative, the aspect ratio requirement will be 3:1, rather than 5:1, unless program-specific sampling and analysis plans (SAPs) specify otherwise or specifically requested otherwise. Thus, fibers (either individual fibers or fibers within disperse matrices or clusters) shall only be recorded if the length is greater than or equal to 0.5 μm and the aspect ratio is greater than or equal to the appropriate criterion. Bundles shall only be recorded if they contain individual constituent fibers with an aspect ratio greater than or equal to the appropriate criterion. The aspect ratio criterion does not apply to compact clusters, compact matrices, or residuals. The overall aspect ratio of a bundle, compact cluster, compact matrix, or residual may have any value.
- c. The definition of a PCM equivalent (PCME) structure is as follows: Any fiber, bundle, matrix, or cluster with an aspect ratio of 3:1 or greater, length longer than 5 μm , and width greater than or equal to 0.25 μm .
- d. The overall dimensions of disperse clusters (CD) and disperse matrices (MD) will not be recorded in two perpendicular directions. The matrix type and individual sub-structures associated with the matrix or cluster will be recorded as described in the ISO method.
- e. Structures that intersect a non-countable grid bar (i.e., top and left grid bars) will be recorded on the count sheet but excluded from the structure density and concentration calculations. These non-countable structures will be denoted with a zero in the Total column.
- f. If a structure originates in one grid opening and extends into an adjacent grid opening, providing that it does not intersect a non-counting grid bar, the entire length of the fiber is recorded.

- g. If a structure intersects both a countable and a non-countable grid bar, the observed length of the structure will be recorded.
- h. See Attachment A for detailed examples of how to record specific structure types that may be encountered in Libby samples.

These modifications and clarifications in structure counting and recording are to provide consistency in analytical procedures and data recording in the project laboratories.

OVERVIEW OF HISTORICAL RECORDING CRITERIA

At the beginning of the Libby project, analytical laboratories (primarily EMSL and RESI) were following the ISO method with regard to structure recording (i.e., recording only those structures meeting an aspect ratio of greater than or equal to 5:1).

Approximately the time of the Phase 2 Investigation (late Spring 2001), project laboratories were instructed by Chris Weis (EPA, Region 8) to record all structures regardless of minimum length or aspect ratio. This recording rule change enabled data users to gain a better understanding of the dimension attributes for structures at the Libby site and allowed for the calculation of PCM equivalent (PCME) structures. In the ISO report generated by the TEM EDD spreadsheet, structures with an aspect ratio less than 5:1 were counted in Bin A and structures with a length less than 0.5 μm were counted in Bin B. Also at this time, the TEM EDD spreadsheet was modified to allow for the capture of the raw structure data, as entered from the laboratory bench sheet, into the Libby site database.

Although it is uncertain exactly when the recording rules changed after the Phase 2 Investigation, based on analyst interviews, project laboratories reverted back to following the ISO method (i.e., recording only those structures meeting an aspect ratio of greater than or equal to 5:1) beginning approximately December 2001, unless specifically requested otherwise in project-specific SAPs and/or QAPPs (e.g., the Supplemental Remedial Investigation samples collected under the SQAPP specified an aspect ratio criterion of greater than or equal to 3:1).

Laboratory modifications LB-000016B through 16F (provided as Attachment B) document the historical laboratory and analyst-specific deviations in recording/counting rules for ISO based on analyst interviews conducted in August and September 2006.

Beginning August 29, 2006, all project laboratories began utilizing an aspect ratio criterion of 3:1, unless specifically requested otherwise.

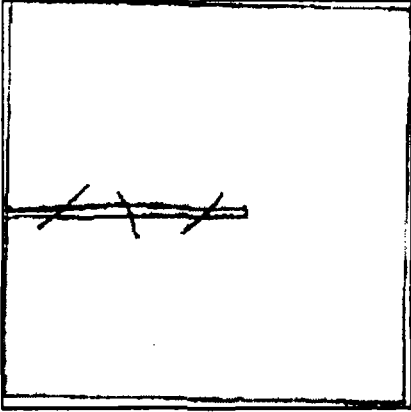
Preparation techniques and recording rules were further refined as part of LB-000053 (effective date: December 21, 2006), whereby all Libby samples were classified as "investigative" and "non-investigative". Samples classified as "investigative" were to utilize an aspect ratio criterion of 3:1, and samples classified as non-investigative were to utilize an aspect ratio criterion of 5:1, unless program-specific sampling and analysis plans (SAPs) specify otherwise or specifically requested otherwise.

Because of the differences in recording rules for ISO analyses across time, data users should be cautious when making comparisons across samples based on the total number of LA structures. The binned metric of total number of LA structures may differ depending upon the recording rule in place at the time.

ATTACHMENT A

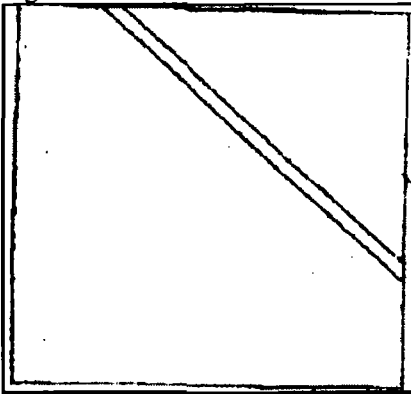
STRUCTURE-SPECIFIC EXAMPLES OF DATA RECORDING

Figure 1



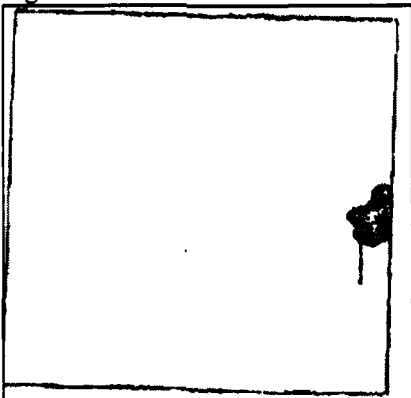
Count as three fibers (F). The large structure is excluded because it crosses a non-countable grid bar (left grid bar).

Figure 2



Count as one fiber (F). Record the length as that observed without doubling.

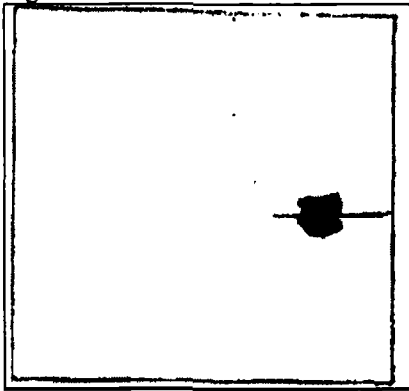
Figure 3



Count as disperse matrix, consisting of one fiber longer than 5 um.

Record as MD11, followed by one fiber (MF). When recording the MF, do not double the length.

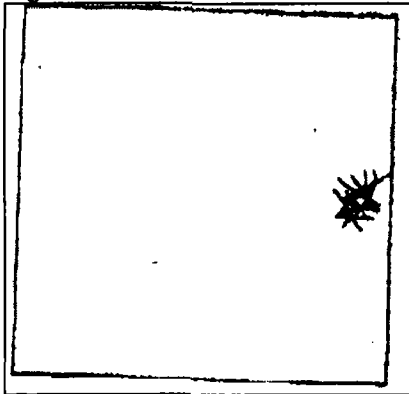
Figure 4



Count as disperse matrix, consisting of one fiber longer than 5 μm .

Record as MD11, followed by one fiber (MF). When recording the MF, double the length of the observed fiber.

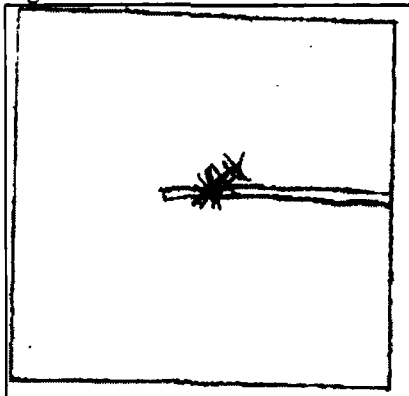
Figure 5



Count as one compact cluster containing more than 9 fibers, which includes one fiber that is longer than 5 μm .

Record as CC+1. When recording the CC, record the length of the cluster as double the length of the observed fiber longer than 5 μm .

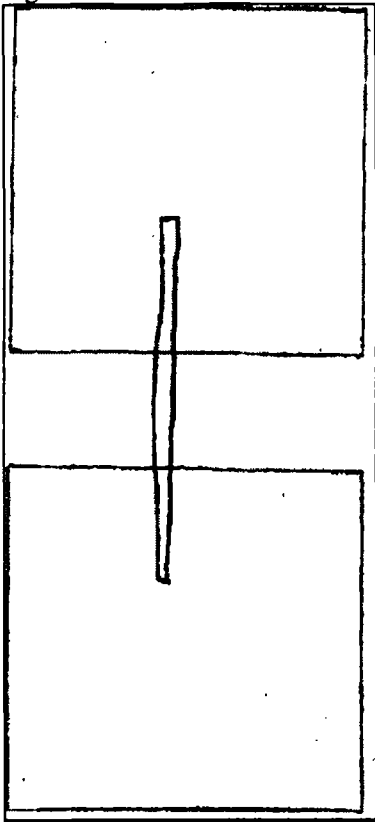
Figure 6



Count as disperse cluster, consisting of one fiber which is longer than 5 μm and one compact cluster residual containing more than 9 fibers.

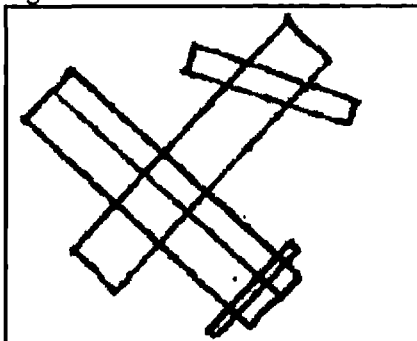
Record as CD+1, followed by one CF and one CR+0. When recording the CF intersecting grid bar, double the length.

Figure 7



Count as one fiber (F). Record the actual length, including protrusion into adjacent grid opening.

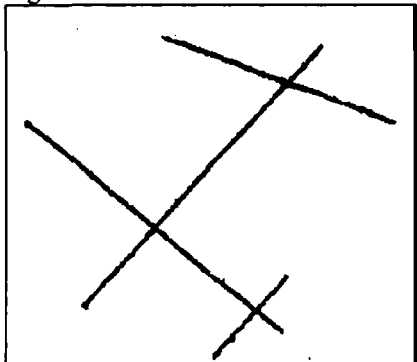
Figure 8



Count as disperse cluster, consisting of four fibers each longer than 5 μm .

Record as CD44, followed by four CFs.

Figure 9



Count as disperse cluster, consisting of four fibers each longer than 5 μm .

Record as CD44, followed by four CFs.

ATTACHMENT B

**LABORATORY AND ANALYST-SPECIFIC DEVIATIONS
IN ISO 10312 RECORDING AND COUNTING RULES PRIOR TO AUGUST 2006
(LB-000016B through 16F)**

**LB-000016B - Batta
LB-000016C - EMSL
LB-000016D - Hygeia
LB-000016E - MAS
LB-000016F - RESI**



Request for Modification
to
Laboratory Activities
LB-000016B

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Bo Li Title: Manager of Microscopy Services
Company: Batta Laboratories, Inc. Date: September 26, 2006

Description of Modification:

Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using ISO 10312, as modified by LB-000016.

Reason for Modification:

To document potential past differences between analysts and laboratories in recording structures using ISO 10312, as modified by LB-000016, and provide a historical timeline of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:

None.

Laboratory Applicability (circle one): All Individual(s) Batta Laboratories, Inc.

This laboratory modification is (circle one): NEW APPENDS to LB-000016A SUPERCEDES _____

Duration of Modification (circle one):

Temporary Date(s): _____
Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable Reject Low Bias Estimate High Bias No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

Beginning May 2002 (when Batta Laboratories began analyzing Libby samples), all analysts recorded structures based on ISO definitions (i.e., aspect ratio criterion of $\geq 5:1$). However, when PCM-equivalent structures were encountered (length $> 5 \mu\text{m}$ and an aspect ratio $\geq 3:1$), they were recorded and marked as countable. The same recording rule was applied to clusters, bundles and matrices that contained PCME structures.

Starting September 11, 2006 (beginning with Lab Job # QC-27, Index ID SQ-00208), recording rules were changed to utilize a length criterion of $> 0.5 \mu\text{m}$ and an aspect ratio criterion of $\geq 3:1$ in accordance with the email direction from Anni Autio (CDM).

DEVIATIONS FROM LAB MODIFICATION LB-000016

Section 4A – Recording of NAM Structures

Analysts were instructed in May 2007 not to record NAMs. However, analysts may occasionally record as NAM if the analyst thought there would be a chance that such a structure might likely be mistaken by a second analyst as a possible asbestos structure. In such a case, "0" was assigned to both primary and total structure columns.

Section 4B – Recording Dimensions for Disperse Clusters (CD) and Matrices (MD)

Dimensions for disperse clusters (CD) and matrices (MD) were measured at two perpendicular directions across the structure. The longest length was defined first, and then the width perpendicular to the length.

Section 4C – Recording Structures Crossing Non-Countable Grid Bars

Before March 2006, structures intersecting the adjacent grid bars on the lower left corner were counted and those intersecting the adjacent bars on the upper right were rejected. After March 2006, this practice was corrected to comply with ISO rules: only those fibers intersecting or touching the lower right corner grid bars were counted and others were rejected.

Section 4D – Recording Structures Crossing Multiple Grid Openings

ISO rules on structures touching grid bars were followed.

Section 4E – Recording Structures Crossing Non-Countable and Countable Grid Bars

Refer to Section 4C above.



Request for Modification
to
Laboratory Activities
LB-000016C

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Ed Cahill Title: National Director
Company: EMSL Analytical, Inc. Date: September 27, 2006

Description of Modification:

Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using ISO 10312, as modified by LB-000016.

Reason for Modification:

To document potential past differences between analysts and laboratories in recording structures using ISO 10312, as modified by LB-000016, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:

None.

Laboratory Applicability (circle one): All Individual(s) EMSL

This laboratory modification is (circle one): **NEW** APPENDS to LB-000016A **SUPERCEDES** _____

Duration of Modification (circle one):

Temporary Date(s): _____
Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Analysts Interviewed

Jodie Bourgerie
Ed Cahill
Robyn Denton
Bob Georgens
Richard Harding
Ken Klutts
Brett Macey
Ron Mahoney

Ex Employees (not interviewed)

Duane Salinas
Thomas Ferrante
Richard White
Anant Samudra
Adrian Arav

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

Beginning in approximately late 1999 (when EMSL Analytical began analyzing Libby samples), all analysts interviewed applied a 5:1 aspect ratio and the counting rules outlined in the ISO method. Sometime during the spring of 2001, verbal directions were given to record all structures, regardless of aspect ratio. Before, during and after this time, if any other aspect ratio than > 5:1 was recorded, it was due to a written or verbal project specific directive (i.e. SQAPP and/or Phase 2 project samples).

DEVIATIONS FROM LAB MODIFICATION LB-000016

All analysts followed ISO method recording rules, as well as the guidance in Laboratory Mod LB-000016 except as noted below:

Section 4A – Recording of NAM Structures

All analysts in compliance.

Section 4B – Recording Dimensions for Disperse Clusters (CD) and Matrices (MD)

All analysts in compliance.

Section 4C – Recording Structures Crossing Non-Countable Grid Bars

Robyn Denton: Has not been adhering to Section 4C which called for recording asbestos structures even when they originated from non-countable grid bars.

Section 4D – Recording Structures Crossing Multiple Grid Openings

All analysts in compliance.

Section 4E – Recording Structures Crossing Non-Countable and Countable Grid Bars

All analysts in compliance.



Request for Modification
to
Laboratory Activities
LB-000016D

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Kyeong Corbin Title: TEM Laboratory Supervisor
Company: Hygeia Laboratories Inc. Date: September 22, 2006

Description of Modification:
Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using ISO 10312, as modified by LB-000016.

Reason for Modification:
To document potential past differences between analysts and laboratories in recording structures using ISO 10312, as modified by LB-000016, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:
None.

Laboratory Applicability (circle one): All Individual(s) Hygeia Laboratories Inc.

This laboratory modification is (circle one): NEW APPENDS to LB-000016A SUPERCEDES _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable Reject Low Bias Estimate High Bias No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

Hygeia Laboratories Inc. received the first batch of ISO 10312 samples on May 30, 2002. Two analysts, Kyeong Corbin and Quynh Trieu, were involved in analyzing all Libby project samples. All ISO samples were counted and recorded for asbestos structures utilizing an aspect ratio criterion of 5:1 or greater, unless the project-specific requirements stated otherwise. For example, all SQAPP samples submitted (first job submitted in June 20, 2005) and all settled dust samples (SRC-Libby-07 method; first job submitted in June 23, 2005) for ISO 10312 method were analyzed for aspect ratio 3:1 or greater.

DEVIATIONS FROM LAB MODIFICATION LB-000016

Both analysts agree on the counting rules mentioned in LB-000016 and clarifications stated below:

1. Hygeia recorded no asbestos detected grid opening as "NSD" instead of "ND" as requested during the period of 6/1/02 to 11/30/02. The Lab Mod LB-000023 was filed.
2. Project-specific stopping rules or target analytical sensitivity superseded the ISO 10312 method stopping rules.

Section 4A – Recording of NAM Structures

NAM structures were not recorded as of August 6, 2002. At times, NAM structures were still recorded if the analysts thought it was necessary and marked as such on the data sheet and on the EDD. (If recorded, NAM structures were noted as non-countable on the data sheet and EDD.)

Section 4B – Recording Dimensions for Disperse Clusters (CD) and Matrices (MD)

All analysts in compliance.

Section 4C – Recording Structures Crossing Non-Countable Grid Bars

All structures intersecting countable grid bars (bottom/south and right/east) were counted and the lengths were doubled. Doubled length was indicated as "X" on the data sheet and indicated as "doubled length" on the comment field of EDD. In general, structures intersecting non-countable grid bars were recorded and indicated as non-countable on the data sheet and EDD. Intersecting grid bar was also indicated on the EDD comment field as crossing E, W, S, N grid bar, i.e. "Non-countable; CWGB," or "CSGB; Doubled length."

Section 4D – Recording Structures Crossing Multiple Grid Openings

All analysts in compliance (see text in Section 4C above).

Section 4E – Recording Structures Crossing Non-Countable and Countable Grid Bars

All analysts in compliance (see text in Section 4C above).



Request for Modification
to
Laboratory Activities
LB-000016E

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Michael D. Mount Title: EM Manager
Company: MAS, LLC Date: December 7, 2006

Description of Modification:
Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using ISO 10312, as modified by LB-000016.

Reason for Modification:
To document potential past differences between analysts and laboratories in recording structures using ISO 10312, as modified by LB-000016, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:
None.

Laboratory Applicability (circle one): All Individual(s) MAS

This laboratory modification is (circle one): **NEW** APPENDS to LB-000016A **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

MAS currently has four analysts (Mike Mount, Kevin Simpson, Mehrdad Motamedi and Will Stark) performing ISO 10312 analyses of Libby samples. The first set of Libby ISO samples (dust samples) were received on 04/03/03. At this time analyst performing analysis were Will Stark, Ding Qian and Jayme Callan. All ISO samples were counted and recorded for asbestos structures utilizing an aspect ratio criterion of 5:1 or greater, unless the project-specific requirements stated otherwise. SQAPP samples and settled dust samples for ISO 10312 submitted since that time (generally around June 2005) methods were analyzed for aspect ratio 3:1 or greater.

DEVIATIONS FROM LAB MODIFICATION LB-000016

All analysts followed ISO method recording rules, as well as the guidance in Laboratory Mod LB-000016 except as noted below:

Section 4A – Recording of NAM Structures

On some occasions one analyst (KS) has recorded non-asbestos structures (NAM) that he deemed significant. However, when NAM structures were recorded, they were always noted as non-countable structures (i.e., placed a "0" in the Total column).

Section 4B – Recording Dimensions for Disperse Clusters (CD) and Matrices (MD)

MAS analysts (MDM and others) may have recorded the overall dimensions of the matrix or cluster rather than the dimensions of the sub-structures.

Section 4C – Recording Structures Crossing Non-Countable Grid Bars

All analysts in compliance. MAS records asbestos structures that are deemed non-countable due to an intersection of the north and west grid bars and places a "0" in the structure count column.

Section 4D – Recording Structures Crossing Multiple Grid Openings

All analysts in compliance. MAS records the entire length of a structure that extends into an adjacent grid opening.

Section 4E – Recording Structures Crossing Non-Countable and Countable Grid Bars

All analysts in compliance. MAS records the entire length of a structure that crosses a non-countable grid bar and a countable grid bar.



Request for Modification
to
Laboratory Activities
LB-000016F

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Jeanne Orr Title: President
Company: RESI Date: December 12, 2006

Description of Modification:
Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using ISO 10312, as modified by LB-000016.

Reason for Modification:
To document potential past differences between analysts and laboratories in recording structures using ISO 10312, as modified by LB-000016, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:
None.

Laboratory Applicability (circle one): All Individual(s) RESI

This laboratory modification is (circle one): NEW APPENDS to LB-000016A SUPERCEDES _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable Reject Low Bias Estimate High Bias No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

Reservoirs Environmental began analyzing samples for the Libby Project in November, 1999. Analysts applied an aspect ratio of 5:1 to the countable structures unless project specific requirements stated otherwise. The samples with a prefix of "SQ-" or "2-" were analyzed with an aspect ratio of 3:1 as requested. Current aspect ratio rules are outlined in LB-000053 and are delineated by the classification of "investigative" or "non-investigative".

DEVIATIONS FROM LAB MODIFICATION LB-000016

All analysts followed ISO method recording rules to the best of their ability, as well as the guidance in Laboratory Mod LB-000016. Samples counted in 1999 and 2000 on historical laboratory bench sheets are identified in LB-000001 through LB-000014.

Section 4A – Recording of NAM Structures

Reservoirs has recorded NAM structures if the analyst determined the structure had morphology similar to an asbestos structure. NAM structures have always been marked as non-countable on the data sheet. The requirement to record non close-call NAM structures such as gypsum or glass was removed after August, 2006.

Section 4B – Recording Dimensions for Disperse Clusters (CD) and Matrices (MD)

All analysts in compliance.

Section 4C – Recording Structures Crossing Non-Countable Grid Bars

All analysts in compliance.

Section 4D – Recording Structures Crossing Multiple Grid Openings

All analysts in compliance.

Section 4E – Recording Structures Crossing Non-Countable and Countable Grid Bars

All analysts in compliance.



Request for Modification
to
Laboratory Activities
LB-000031A

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): ☒ TEM-AHERA ☐ TEM-ISO 10312 ☐ PCM-NIOSH 7400 ☐ NIOSH 9002
EPA/600/R-93/116 ☒ ASTM D5755 ☐ EPA/540/2-90/005a ☐ SRC-LIBBY-03
Other: _____

Requester: Lynn Woodbury Title: Technical Consultant
Company: Syracuse Research Corporation Date: January 18, 2008

Description of Modification:
Permanent modifications and clarifications to the Transmission Electron Microscopy analysis of air samples using AHERA and dust samples using ASTM. The purpose of the attached is to document historic modifications & clarifications, and provide additional permanent clarifications.

Reason for Modification:
To optimize the efficiency of air and dust sample analysis and to provide consistency in analytical procedures and data recording in the project laboratories.

Potential Implications of this Modification:
Modifications reflect changes necessary to clarify ISO requirements in relation to project-specific issues. Negative implications - comparisons of the Total # of LA structures between historical results and current results may be biased (high or low) due to differences in recording rules with regard to aspect ratio criteria. Positive implications - consistency in procedures between and within project laboratories and documentation of those procedures.

Laboratory Applicability (circle one): ☒ All Individual(s) _____

This laboratory modification is (circle one): **NEW** APPENDS to _____ ☒ SUPERCEDES LB-000017, LB-000017A, LB-000031

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

☒ Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable Reject ☒ Low Bias Estimate ☒ High Bias No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

AHERA/ASTM MODIFICATIONS AND CLARIFICATIONS

1. Overloading Modification:

A rejection criteria of >25% obscuration will be used for this project. The 25% overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, Camp Dresser McKee (CDM), Volpe Center, and Reservoirs (RESI). [The AHERA method grid rejection of 10% has been established and clarified by NVLAP Lab Bulletin 7-2002 – July 12, 2002].

2. Indirect Preparation Of Air Samples Modification:

If the sample is visibly overloaded or contains loose debris, it will be prepared indirectly according to procedures provided in SOP EPA-Libby-08. Secondary filters will be analyzed according to the AHERA/ASTM counting rules for this project. Calculations will be adjusted to contain a dilution factor. This indirect preparation procedure will enable the capture of data from samples that otherwise would be rejected.

3. Stopping Rule Clarification:

For this project, a maximum of ten grid openings will be read unless specifically instructed otherwise.

4. Abundant Chrysotile Modification:

If abundant chrysotile is present, the chrysotile count may be terminated in accordance with the counting rules specified in LB-000039.

5. Cassette Modification:

Cassettes with a 0.8 µm pore size and no 5.0 µm diffuser filter (PCM cassettes) are primarily used for AHERA/ASTM sample collection.

6. Structure Counting and Recording Modifications and Clarifications:

- a. All structures will be recorded with the following "Structure Type" designations on the EPA Region 8 spreadsheet:

Fiber	F
Bundle	B
Cluster	C
Matrix	M

If the analyst determines that additional information is needed to describe a structure, comments pertaining to the structure in addition to a sketch will be recorded in the structure comments column.

- b. Non-asbestos material (NAM) structures are not being recorded, unless identified as a "close call" (see LB-000066 for details). This project-specific modification stems from the need only to quantify levels of contaminants of concern (i.e., asbestos) at a given sample location.
- c. For the Libby project, a designation of "ND" (none detected) will be used to document when no structures are detected for the grid opening.
- d. Recording rules will be as described in the AHERA method except that the aspect ratio requirement will depend upon the classification of the sample as "investigative" or "non-investigative", as specified in LB-000053. If samples are classified as investigative, the aspect ratio requirement will be 3:1, rather than 5:1, unless program-specific sampling and analysis plans (SAPs) specify otherwise or specifically requested otherwise. Thus, fibers shall only be recorded if the aspect ratio is greater than or equal to the appropriate criterion. Bundles, clusters, and matrices shall only be recorded if they contain individual constituent fibers with an aspect ratio greater than or equal to the appropriate criterion. The overall aspect ratio of a bundle, cluster, or matrix may have any value.
- e. Structures that are non-countable (e.g., aspect ratio does not meet the appropriate criterion, matrices without an

exposed termination) should be recorded for informational purposes, but identified as non-countable, to ensure they are excluded from structure density and concentration calculations. These non-countable structures will be denoted with a zero in the Total column.

- f. If a structure originates in one grid opening and extends into an adjacent grid opening, the entire length of the fiber is recorded.
- g. The AHERA method requires the analyst to "record the length category and structure type classification non the count sheet after the field number and fiber number". As a clarification to this, the actual length and width of individual fibers, bundles, compact clusters, and compact matrices will be recorded. For disperse clusters and matrices, the length of only the longest protruding structure will be recorded. It is not appropriate to record all sub-structures as in the ISO 10312 method. Structure dimensions may be recorded in microns (μm) or screen units provided that the scaling factors are recorded. See Attachment A for detailed examples of how to record specific structure types that may be encountered in Libby samples.
- h. In the AHERA method, one of the provided illustrations for bundles is unclear (i.e., the bundle appears as three fibers). The AHERA written definition for bundles will be utilized.

These modifications and clarifications in structure counting and recording are to provide consistency in analytical procedures and data recording in the project laboratories.

OVERVIEW OF HISTORICAL RECORDING CRITERIA

At the beginning of the Libby project, analytical laboratories (primarily EMSL and RESI) were following the AHERA method with regard to structure recording (i.e., recording only those structures meeting an aspect ratio of greater than or equal to 5:1).

Approximately the time of the Phase 2 Investigation (late Spring 2001), project laboratories were instructed by Chris Weis (EPA, Region 8) to record all structures regardless of minimum length or aspect ratio. This recording rule change enabled data users to gain a better understanding of the dimension attributes for structures at the Libby site and allowed for the calculation of PCM equivalent (PCME) structures.

Although it is uncertain exactly when the recording rules changed after the Phase 2 Investigation, based on analyst interviews, project laboratories reverted back to following the AHERA method (i.e., recording only those structures meeting an aspect ratio of greater than or equal to 5:1) beginning approximately in late 2001 to early 2002.

Laboratory modifications LB-000031B through 31F (provided as Attachment B) document the historical laboratory and analyst-specific deviations in recording/counting rules for AHERA/ASTM based on analyst interviews conducted in August and September 2006. Beginning August 29, 2006, all project laboratories were instructed to utilize an aspect ratio criterion of 5:1 for AHERA/ASTM analyses, unless specifically requested otherwise.

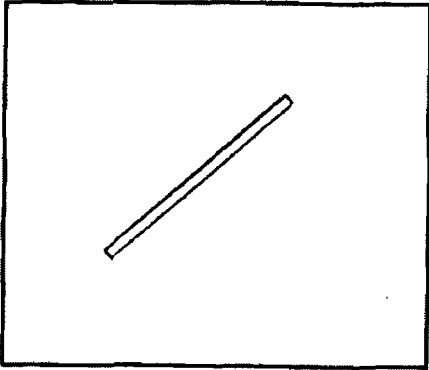
Beginning April 24, 2007, all project laboratories were instructed to utilize an aspect ratio criterion of 3:1 for AHERA/ASTM analyses of "investigative" samples. Instruction regarding classification of samples as "investigative" and "non-investigative" was provided as part of LB-000053 (effective date: December 21, 2006). Samples classified as "non-investigative" were to utilize an aspect ratio criterion of 5:1, unless program-specific sampling and analysis plans (SAPs) specify otherwise or specifically requested otherwise.

Because of the differences in recording rules for AHERA/ASTM analyses across time, data users should be cautious when making comparisons across samples based on the total number of LA structures. The binned metric of total number of LA structures may differ depending upon the recording rule in place at the time.

ATTACHMENT A

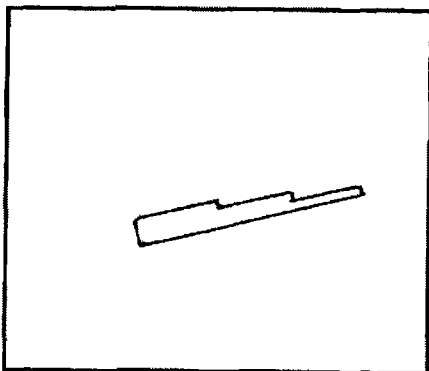
STRUCTURE-SPECIFIC EXAMPLES OF DATA RECORDING

Structure 1:



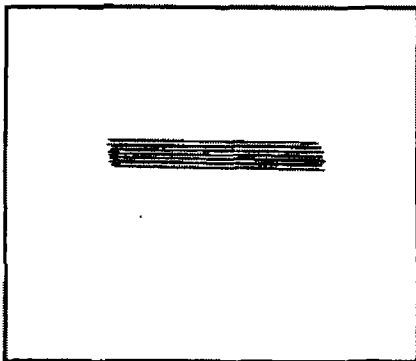
Simple fiber – Record length and width. Structure must meet AHERA length and aspect ratio criteria.

Structure 2:



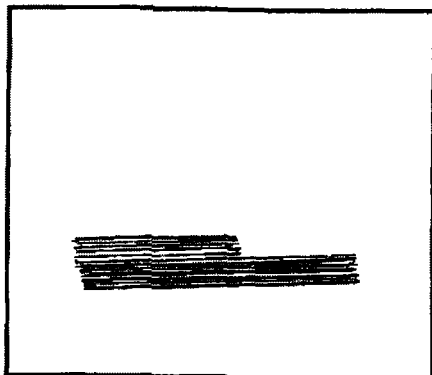
Stepped fiber – Record length. Record width as a best estimate of the average width. Structure must meet AHERA length and aspect ratio criteria.

Structure 3:



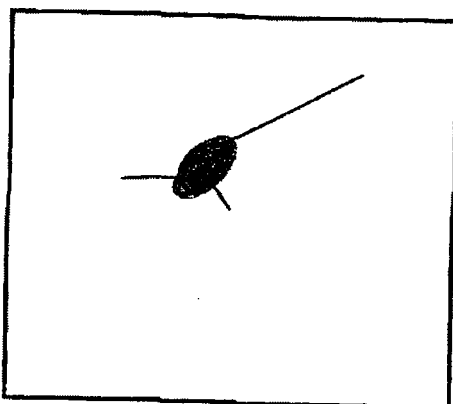
Bundle – Record length and width of bundle. The aspect ratio of the overall structure is not a factor. At least three individual sub-structures in parallel arrangement separated by less than one sub-structure diameter, adequate to meet AHERA bundle definition, must meet AHERA length and aspect ratio criteria.

Structure 4:



Stepped bundle – Record longest length of bundle. Record width as a best estimate of the average width. The aspect ratio of the overall structure is not a factor. At least three individual sub-structures in parallel arrangement separated by less than one sub-structure diameter, adequate to meet AHERA bundle definition, must meet AHERA length and aspect ratio criteria.

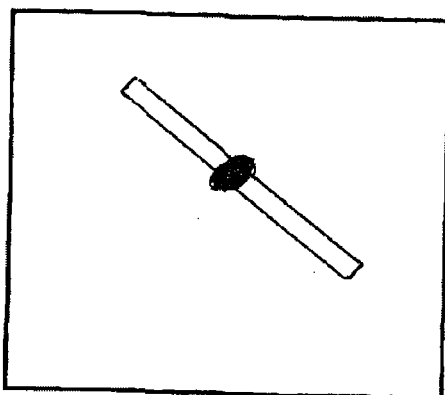
Structure 5:



Matrix – Record length of longest exposed structure and its width. Structure must meet AHERA length and aspect ratio criteria.

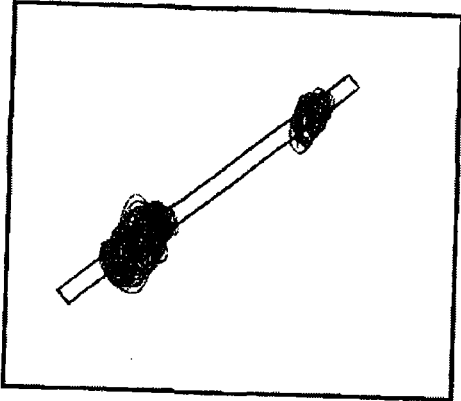
[Do not record the dimensions of the matrix, only the longest protruding structure.]

Structure 6:



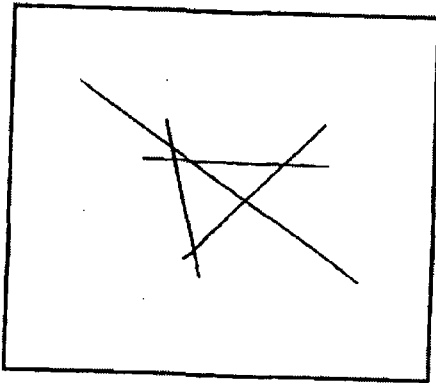
Fiber with adhering matrix material - This structure does not fall into the matrix category as defined in that both ends are exposed (definition 14, AHERA) - Record length and width of the fiber. Structure must meet AHERA length and aspect ratio criteria.

Structure 7:



Structure with protrusions $<$ aspect ratio criterion but an overall aspect ratio meeting criterion. Provided that the structure can be observed to be continuous through the adhering material, count as a fiber. Structure must meet AHERA length and aspect ratio criteria. If the structure cannot be observed to be continuous through the adhering material, do not count.

Structure 8:



Cluster – Record the length of the longest observable sub-structure. Record width as a best estimate of the average width of the overall structure, not the individual sub-structures. The aspect ratio of the overall structure is not a factor. There must be at least three intersections comprised of individual sub-structures that meet AHERA length and aspect ratio criteria to meet cluster definition.

ATTACHMENT B

**LABORATORY AND ANALYST-SPECIFIC DEVIATIONS
IN AHERA/ASTM RECORDING AND COUNTING RULES PRIOR TO AUGUST 2006
(LB-000031B through 31F)**

**LB-000031B - Batta
LB-000031C - EMSL
LB-000031D - Hygeia
LB-000031E - MAS
LB-000031F - RESI**



Request for Modification
to
Laboratory Activities
LB-000031X

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Bo Li Title: Manager of Microscopy Services
Company: Batta Laboratories, Inc. Date: September 26, 2006

Description of Modification:
Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031.

Reason for Modification:
To document potential past differences between analysts and laboratories in recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:
None.

Laboratory Applicability (circle one): All Individual(s) Batta Laboratories, Inc.

This laboratory modification is (circle one): **NEW** APPENDS to LB-000031A **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

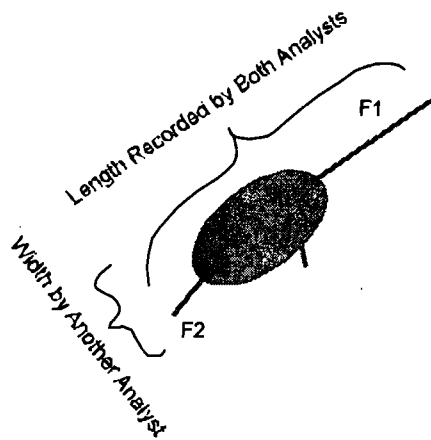
Beginning May 2002 (when Batta Laboratories began analyzing Libby samples), all analysts used fiber definition of $\geq 5:1$ aspect ratio and $\geq 0.5 \mu\text{m}$ length for all AHERA analyses, and any project-specific requirements regarding the aspect ratio were incorporated into AHREA/ASTM (dust) analyses as available through various lab modifications.

DEVIATIONS FROM LAB MODIFICATIONS LB-000017, LB-000017A, LB-000031

Prior to LB-000066, NAM structures were recorded by all analysts when deemed necessary by the analysts. Inconsistencies regarding the measurement and recording of clusters and matrices began as early as May, 2003 when compared with the guidelines outlined in LB-000031). See Attachments #1 through #4 for clarifications and illustrations. Figure numbers in the following attachments are corresponding to structure examples provided in LB-000031.

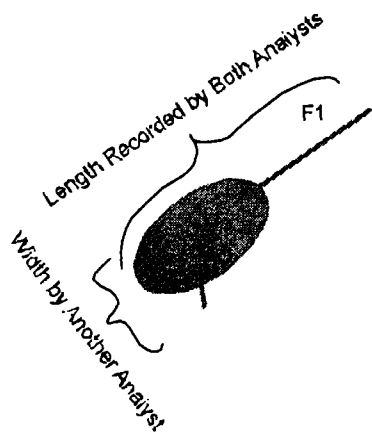
Batta Laboratories, Inc. began to comply with LB-000031 among analysts beginning August 23, 2006, with sample batch CDM-91 (EPA Job# L10972).

Figure 5: Matrix



Batta recorded as one matrix:

width: one analyst took the average of F1 and F2 (if both are within the fiber definition), and the other took the overall width of the entire structure as illustrated.

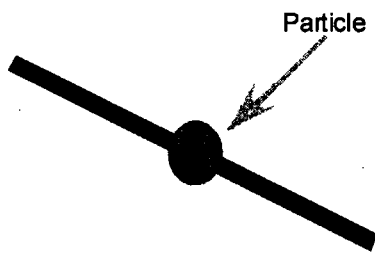


Batta recorded as one matrix:

width: refer to above.

Attachment #2

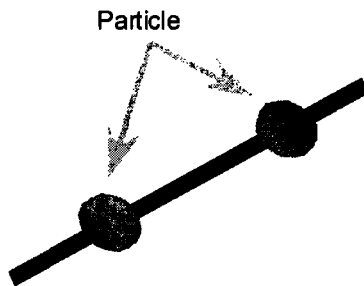
Figure 6



Batta recorded as either as one fiber or one matrix:

- 1) Matrix: particle size is equal or greater than 0.5 micron.
- 2) Fiber: particle size is less than 0.5 micron.

Figure 7

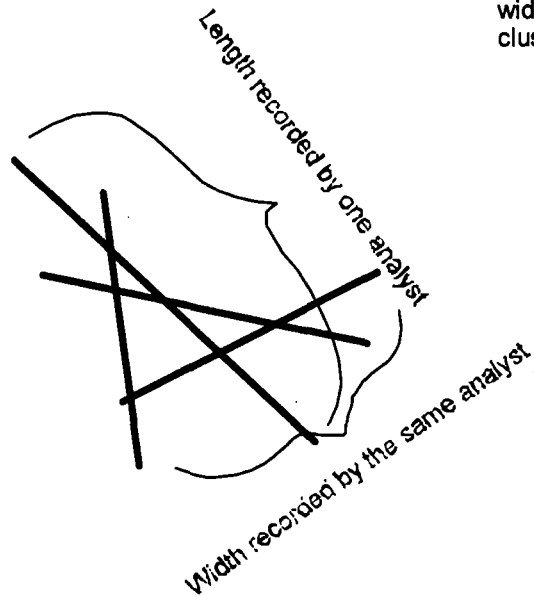


Batta recorded as either a fiber or a matrix:

- 1) Fiber: both particles are less than 0.5 micron
- 2) Matrix: any one of the particles is equal or greater than 0.5 micron
- 3) Not a structure: both exposed fiber ends do not meet the fiber definition and both particles are equal or greater than 0.5 micron

Attachment #3

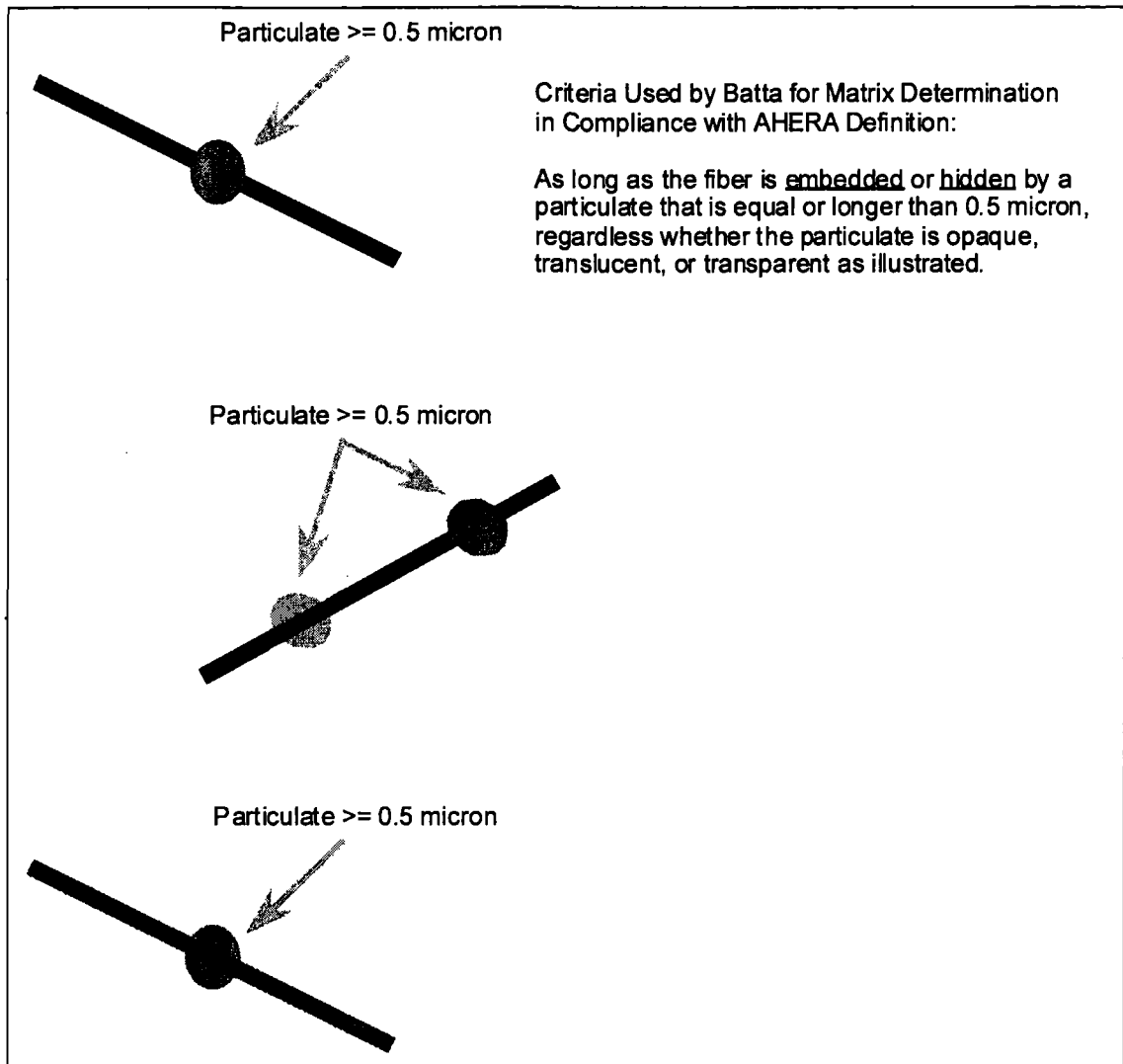
Figure 8: Cluster



Batta has internal discrepancies in recording the dimension of the this structure:

One analyst recorded as illustrated; the other recorded as the following:
length = the longest fiber within the cluster
width = average width of all fibers within the cluster

Attachment #4





Request for Modification
to
Laboratory Activities
LB-000031C

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
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All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Ed Cahill Title: National Director
Company: EMSL Analytical Date: September 26, 2006

Description of Modification:
Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031.

Reason for Modification:
To document potential past differences between analysts and laboratories in recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:
None.

Laboratory Applicability (circle one): All Individual(s) EMSL Analytical

This laboratory modification is (circle one): **NEW** APPENDS to LB-000031A **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Analysts Interviewed

Steve Bennett
Jodie Bourgerie (August 2001 in Libby and 5 times after)
Ed Cahill
Joe Centifonti
Robyn Denton (late 2002 and 2003)
Bob Georgens
Richard Harding
Ken Klutts
Brett Macey
Ron Mahoney
Anant Samudra
Paul Senne (in Libby 2003, 2004, 2005)

Ex Employees (not interviewed)

Adrian Arav (whereabouts unknown)
Duane Salinas (whereabouts unknown)
Tom Ferrante (deceased)
Richard White (whereabouts unknown)

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

Beginning in late 1999, the aspect ratio applied by EMSL was greater than or equal to 5:1. Sometime in the spring of 2001, analysts received verbal direction to record structures regardless of aspect ratio. In late 2001 or early 2002, project analysts reverted back to 5:1. EMSL analyst interviews were conducted in August and September 2006. On August 29, 2006, all laboratories were instructed to use 5:1 for AHERA/ASTM analyses.

DEVIATIONS FROM LAB MODIFICATIONS LB-000017, LB-000017A, LB-000031

All analysts followed AHERA/ASTM method recording rules, as well as the guidance in the above laboratory modifications, except as noted below. Figure numbers in the following attachments are corresponding to structure examples provided in LB-000031.

structure	Steve Bennett
2	record longest L and maximum width
3	as stated in Mod 31
4	record longest L and maximum width
8	record longest sub-structure L and maximum width perpendicular to that sub-structure

structure	Joe Centifonti
2	Maximum width
3	As stated in Mod 31, BUT AR of whole bundle is used as criterium
4	Maximum width
5	Add ALL fiber Lengths and average fiber width
6	As stated in Mod 31, BUT Matrix
7	As stated in Mod 31, BUT Matrix
8	L=longest fiber BUT W=average width

structure	Robyn Denton (late 2002 and 2003)
5	Pre 2005 L=add up all sub structure L. W=average W of sub structure fibers. From 2005 on, overall L and W was recorded.
6	Matrix. Pre 2005 L=add up all sub structure L W=average W of sub structure fibers. From 2005 on overall L and W was recorded.
7	would call Matrix

structure	Bob Georgens
2	record longest L and maximum width
4	record longest L and maximum width

- 6 matrix not fiber. Record total L and max width
- 7 matrix not fiber. Record total L and max width
- 8 record longest sub-structure L and widest sub-structure W

structure	Ken Klutts
3	As stated in Mod 31, UNLESS AR of bundle <5 the record average width of component fibers
4	As stated in Mod 31, UNLESS AR of bundle <5 the record average width of component fibers
5	Add ALL AHERA countable fiber Lengths and average fiber width
8	L = longest fiber W = average fiber width

structure	Brett Macey
3	as stated in Mod 31 BUT AR of whole bundle is used as criterion
5	Add ALL fiber Lengths and average fiber width
6	as stated in Mod 31 BUT Matrix
7	as stated in Mod 31 BUT Matrix
8	L=longest fiber BUT W=maximum width

structure	Anant Samudra
8	record longest sub-structure L and maximum width perpendicular to that sub-structure <i>**At times, recorded AHERA analyses in ISO 10312 format</i>

structure	Paul Senne (in Libby 2003, 2004, 2005)
6	Matrix if matrix material is >0.5 microns



Request for Modification
to
Laboratory Activities
LB-000031D

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Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Kyeong Corbin Title: TEM Laboratory Supervisor
Company: Hygeia Laboratories Inc. Date: September 20, 2006

Description of Modification:
Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031.

Reason for Modification:
To document potential past differences between analysts and laboratories in recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:
None.

Laboratory Applicability (circle one): All Individual(s) Hygeia Laboratories Inc.

This laboratory modification is (circle one): **NEW** APPENDS to LB-000031A **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
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Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

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Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

Hygeia Laboratories Inc. received the first batch of AHERA samples on September 16, 2002. Two analysts, Kyeong Corbin and Quynh Trieu, were involved in analyzing all Libby project samples. All AHERA samples were analyzed according to the AHERA counting rules (aspect ratio 5:1 or greater) and any project-specific requirements were incorporated as available. At times, asbestos structures with an aspect ratio of at least 3:1 were recorded by the analyst. These were indicated as such on the data sheet and on the EDD, and were recorded as non-countable (i.e., total column populated with a zero).

DEVIATIONS FROM LAB MODIFICATIONS LB-000017, LB-000017A, LB-000031

Both analysts agree on the counting rules mentioned in LB-000017, LB-000017a, and LB-000031 and clarifications stated below:

1. Hygeia recorded no asbestos detected grid opening as "NSD" as expressed in the AHERA instead of "ND" as requested by project modification during the period of 6/1/02 to 11/30/02. The Lab Mod LB-000023 was filed.
2. As results of August 6, 2002 teleconference, the following instructions were given to the analysts:
 - Do not count NAM structures. At times, NAM structures were still recorded if the analyst thought it was necessary and marked as such on the data sheet and on the EDD, and were recorded as non-countable (i.e., total column populated with a zero).
 - Record fiber, bundle, cluster, and matrix as F, B, C, M as indicated by AHERA. (Do not record matrix as MF, MB, MC, F/M, B/M, or C/M.)
3. As results of August 13, 2002 teleconference, the following instructions were given to the analysts:
 - Measure only visible portion of the fiber or bundle sticking out of the matrix. (Prior to this date, any matrix components that were embedded in opaque matrix were measured by doubling length or to the end of matrix depending on the matrix size.)
 - It is a non-countable structure if both ends were embedded in the matrix. (In general, non-countable structures were recorded and indicated as such on the data sheet and on the EDD.)
 - Intersection is defined as stated on the AHERA, i.e. Non-parallel toughing or crossing of fibers, with the projection having an aspect ratio 5:1 or greater. (If <5:1 projection, it was considered no intersection.)
4. As results of September 9 and September 13, 2003 teleconferences, the following clarifications were given to the analysts as of October 2003:
 - No doubling of length measurement when crossing grid bars (different from ISO, i.e. ISO requires length to be doubled);
 - Count all grid bars;
 - Measure visible portion only;
 - For C or M, record the dominant/longest sub-structure's dimension;
 - Do not use F/m, B/m, or C/m classification; just record it as M, matrix.
5. Stopping rules for chrysotile structures were changed from 100 structures to 50 structures in October 2003. The laboratory was instructed to file an individual lab mod for these cases.



Request for Modification
to
Laboratory Activities
LB-000031E

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Michael D. Mount Title: EM Manager
Company: Materials Analytical Services Date: September 25, 2006

Description of Modification:
Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031.

Reason for Modification:
To document potential past differences between analysts and laboratories in recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:
None.

Laboratory Applicability (circle one): All Individual(s) Materials Analytical Services

This laboratory modification is (circle one): **NEW** APPENDS to LB-000031A **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

MAS currently has four analysts (Mike Mount, Kevin Simpson, Mehrdad Motamedi, and Will Stark) performing ISO 10312 analyses of Libby samples. The first set of Libby AHERA samples (dust samples) were received on 10/08/04. At this time, analysts performing TEM analysis were Will Stark, Ding Qian and Kevin Simpson. AHERA/ASTM method dust samples were counted and recorded for asbestos structures utilizing an aspect ratio criterion of 5:1 or greater, unless the project-specific requirements stated otherwise.

DEVIATIONS FROM LAB MODIFICATIONS LB-000017, LB-000017A, LB-000031

All analysts followed AHERA/ASTM method recording rules, as well as the guidance in the above laboratory modifications, except as noted below:

- Between 1/1/06 to 8/31/06, TEM analysts Mike Mount and Mehrdad Motamedi, recorded asbestos fibers and bundles with visible ends as a matrix structure instead of a fiber or bundle.



Request for Modification
to
Laboratory Activities
LB-000031F

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Jeanne Orr Title: President
Company: Reservoirs Environmental, Inc. Date: September 25, 2006

Description of Modification:
Laboratory-specific clarification of potential inconsistencies among analysts when recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031.

Reason for Modification:
To document potential past differences between analysts and laboratories in recording structures using AHERA/ASTM, as modified by LB-000017, LB-000017A, and LB-000031, and provide a historical timeline for each Libby laboratory of potential changes in aspect ratio recording rules.

Potential Implications of this Modification:
None.

Laboratory Applicability (circle one): All Individual(s) Reservoirs Environmental, Inc.

This laboratory modification is (circle one): **NEW** APPENDS to LB-000031A **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: HISTORIC
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable Reject Low Bias Estimate High Bias No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

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No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

HISTORIC INFORMATION ON ASPECT RATIO RECORDING RULES

Reservoirs Environmental began analyzing samples for the Libby Project in November, 1999. Analysts applied an aspect ratio of 5:1 to the countable structures unless project specific requirements stated otherwise.

DEVIATIONS FROM LAB MODIFICATIONS LB-000017, LB-000017A, LB-000031

All analysts followed AHERA/ASTM method recording rules, as well as the guidance in the above laboratory modifications, except as noted below:

- Analysts at Reservoirs have historically characterized a fiber with both ends visible and matrix in the middle as a matrix; whereas LB-000031 designates this type of structure as a fiber.
- Samples counted in 1999 and 2000 were recorded on historical laboratory bench sheets and are identified in LB-000001 through LB-000014.
- In August, 2002 the project no longer required that NAM structures be recorded. On occasion, analysts recorded NAM structures when the analyst found the structure notable. The NAM structure was noted as non-countable by recording a zero in the total column.
- Excluded structures, such as a fiber with both ends obscured by matrix or a fiber with less than the required aspect ratio, were recorded and designated as non-countable by recording a zero in the total column.